

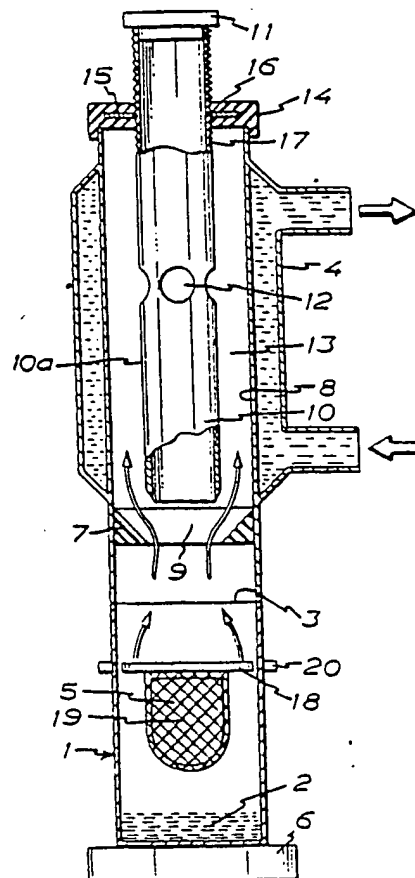
INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(54) Title: METHOD AND APPARATUS FOR THE RECOVERY OF SOLVENT FROM A SOLUTION, IN PARTICULAR AN EXTRACT, AND FOR STORING THE RECOVERED SOLVENT

(57) Abstract

In a method and an apparatus for the recovery of solvent from a solution and for storage of the recovered solvent, solvent vapour obtained by distillation is led to a condenser (4) by means of a conduit (10) movably disposed in the longitudinal direction of the condenser, the outer wall (10a) of the conduit, by shifting of the conduit in one direction, may be brought to sealingly close the condenser against vapour condensate loss in a direction against the vapour flow. On shifting of the conduit (10) in the other direction, the condenser (4) is opened for discharging vapour condensate. The method and the apparatus may be used in extraction processes, for example fat determination analysis, for rapid and substantially loss-free reuse of the solvent.



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METHOD AND APPARATUS FOR THE RECOVERY OF SOLVENT FROM
A SOLUTION, IN PARTICULAR AN EXTRACT, AND FOR STORING
THE RECOVERED SOLVENT

The present invention relates to a method and an apparatus for the recovery of solvent from a solution and for storing the recovered solvent for rapid re-use thereof.

5 For extraction processes on a laboratory scale for, for example, analysis, use is often made of a flask or container with a condenser which may be sealingly connected at the top of the container. A filter thimble or crucible accommodating a sample which is to be ex-
10 tracted is inserted into the container which contains solvent or extractor for sample component parts, and the solvent is heated up to boiling point. The solvent vapours condense in the condenser and fall back into the container. The filter crucible with the sample may
15 be immersed in the solvent or be located above the level of the solvent, in which latter case the solvent vapour and condensed solvent vapour or vapour condensate constitute the extractor.

In such extraction for analysis or for further processing of extracted sample component parts, the solvent
20 is often distilled off from the extract. The analysis may, in such a case, relate, for example, to weighing. An example of such a process is fat determination in accordance with the Soxhlet process. The distillation
25 is often carried out with a loss of the solvent or utilizing special distillation apparatuses with receivers for taking care of the solvent. However, a certain amount of the solvent is lost also in this latter case, more precisely in the components of the
30 distillation apparatus.

The object of the present invention is to realize a method and an apparatus which eliminates the above-mentioned disadvantages and which in a simple and



effective manner makes possible the recovery and storage of solvent and the rapid re-use thereof.

This is achieved according to the invention by means of a method and an apparatus having the characteristic features disclosed in the claims.

In brief, the basic concept of the invention is that the solvent from an extract or other solution is vaporized and led via a conduit, which is axially movably disposed in a condenser, to a space located outside the conduit but essentially within the condenser, the space including the cooling surfaces of the condenser and being, at one end, sealable by means of the outer wall of the conduit by an axial shifting of the conduit to this end, the end being, furthermore, connectable to a container for the solution. The distilled-off or vaporized solvent departs, thus, from the container to the space via the conduit and is retained in this space until the conduit is retracted from the above-mentioned space end, in which event the recovered solvent may run into the container for re-use.

The invention is particularly favourably suited to extraction processes, in particular extraction for the purposes of analysis, since it permits rapid mutually subsequent extraction cycles. However, the invention is generally applicable to all types of solvent recovery processes in which it is desirable to be able rapidly to re-use the solvent without any appreciable losses.

The invention will be described in greater detail hereinbelow with reference to the accompanying drawing in which Fig. 1 shows, partly in longitudinal section, an extraction apparatus including a recovery apparatus according to the invention and Fig. 2 shows a cut-away portion of the apparatus of Fig. 1 in a condition of readiness for the recovery of solvent.

A container 1 for extractor or solvent/extract 2 is releasably connected at 3 to a vertical recycle

cooler or condenser 4. A filter crucible or thimble 5, arranged to contain the solid sample intended for extraction, is inserted in the container 1 and a heater 6 is provided for heating the solvent 2 to its boiling point.

The hitherto described extraction apparatus is conventional in laboratories and has a conventional function, for which reason no detailed description of this apparatus is necessary. It should be emphasized that the component parts of the apparatus may each be of conventional type. Thus, the recycle cooler or condenser 4 may be of the ball type, spiral type or, as shown on the drawing, of the Liebig type, with paths for liquid cooling, or without such paths if the extraction is to be carried out under air cooling. The important feature is that the apparatus allows of the mounting-in and operation of a conduit (which will be described below) shiftable in its longitudinal direction and forming part of an apparatus according to the invention for the recovery of solvent. The releasable connection at 3 between the container 1 and the condenser 4 may, if the container 1 advantageously has the same inner diameter as the lower end of the condenser 4, be of the type disclosed in our Swedish Patent No. 7611015-4 or in U.S. patent application No. 877,959, filed on February 15, 1978 and assigned to Tecator Instrument AB. The releasable connection at 3 may also be defined by a sealing plug through which the lower end of the condenser is passed, or by a ground end of the container 1 for receiving a ground lower end of the condenser 4. Furthermore, the filter crucible or thimble 5 may, if it consists of easily collapsible material, such as paper, be supported in a conventional manner by a metal wire mesh basket which keeps the crucible 5 immersed in the solvent 2 or above the level of the solvent 2 in the container 1. If the extraction process is to be carried out only by means



of vapour and vapour condensate, the crucible 5 may be supported in a conventional Soxhlet extractor which has two parallel-coupled conduits, of which one serves to lead the solvent vapour to the condenser, whereas the other serves to receive the filter crucible and the vapour condensate reflowing from the condenser and also serves to lead the vapour condensate further to the container 1 for solvent/extract.

An apparatus for the recovery and storage of solvent or extractor after completed extraction comprises the condenser 4 and, in the illustrated embodiment, a sealing bushing 7 of resilient material which is disposed within a lower end portion of the condenser at a position where the condensation surface 8 of the condenser is not yet capable to any great extent of condensing the solvent vapour rising in the condenser, that is to say at a position which the solvent vapour must first pass on its upward travel before any appreciable condensation of the vapour takes place. In the relaxed state, the bushing 7 has a slightly greater outer circumference than the inner circumference of the lower end of the condenser 4 where the bushing is placed, so that good retention and sealing forces are obtained against the inner wall of the condenser once the bushing has been inserted in place through the open lower end of the condenser. An inner wall of the bushing forming a circular opening 9 is also disposed, when the bushing is in place, to sealingly engage with the outer wall 10a of a circular conduit 10 which is shiftable in its axial direction within the condenser a distance inside the condensation surfaces thereof (that is to say in, for example, a spiral condenser with cooling agent flow in the spiral radially inwardly of the spiral turns), when the conduit is lowered from an upper position (Fig. 1) in which the outside of the conduit is not in engagement with the inner wall of the bushing, to a lower position

(Fig. 2). As shown on the drawing, the inner wall of the bushing advantageously conically tapers downwardly towards the container 1, such that the conical wall together with the lower surface of the bushing, forms a lip sealing against the conduit 10 when the conduit engages with the bushing, whereby the bushing also forms a lower guide for the conduit 10. The material of the bushing 7 and the conduit 10 are selected such that they withstand the prevalent vapour temperatures.

At its upper end, which projects beyond the upper end of the condenser 4, the conduit 10 is sealed by means of a plug 11 and the conduit 10 has a number of orifices 12 in its wall distributed in a circumferential direction at a region of its length surrounded by the condensation or cooling surface 8 of the condenser 4, such that the interior of the conduit 10 is in communication with a space 13 which is laterally defined by the outside of the conduit 10 and the condensation surface 8. This space is above the level of the orifices 12, when the conduit is in the upper position, partially sealed by means of a collar 14 which serves as an upper guide for the conduit and has passages for connecting the space 13 with the atmosphere. A metal washer 15 embedded in the collar 14 has an inwardly projecting pin 16 which engages in a helical groove 17 in the outside of the conduit 10, such that, on rotation of the conduit 10 at its upper end, the conduit may be moved from the upper position shown in Fig. 1 to the lower position shown in Fig. 2, and vice versa. Naturally, other possibilities are obvious to a person skilled in the art for maintaining the conduit in the upper position, in which event the shifting of the conduit may take place parallel with generatrix on the conduit.

The smaller end of the opening 9 may advantageously be given the same diameter as the crucible 5, such that



if the crucible, after a completed extraction process with the hot solvent is lifted above the level of the solvent/extract 2 in the container 1 for continued extraction with solvent vapour and vapour condensate, substantially all of the vapour condensate may run down into the crucible. Raising and lowering of the crucible may be effected by means of, for example, a magnet arrangement illustrated in principle in Fig. 1, in which an annular magnet 19 within the container 1 fixedly retains the crucible 5 which, in such an instance, is mounted on or in a magnetisable metal portion, for example, a steel wire mesh basket 19 and may be influenced by means of another semi-annular magnet 20 outside the container for upward and downward movement.

The function of the extraction apparatus illustrated in Figs. 1 and 2 is as follows. On heating of the solvent 2 by means of the heater 6 for extraction of the sample in the crucible 5, the solvent vapour rises, as shown by means of arrows in Fig. 1, up in the condenser 4 through the opening 9 and falls, after condensation, through the opening 9 back into the container 1 and/or into the crucible 5. After completed extraction of the sample, the conduit 10 is rotated at its upper end so that it moves downwardly to the lower position where the outer wall of the conduit sealingly engages with the bushing 7, the space 13 being shut-off at its bottom so that, on distillation-off of the solvent from the extract in the container, the solvent vapour can only leave the container 1 through the conduit 10, as shown by means of an arrow in Fig. 2. The vapour leaves the conduit via the orifices 12, is condensed by the cooling surfaces 8 and collected in the space 13 beneath the orifices 12, recycling to the container being prevented by the sealing engagement between the conduit 10 and the bushing 7 (please see Fig.2). After distillation-off of the solvent, the connection at 3 may be

released and the crucible 5 with the ready-extracted sample may be removed from the container 1 and the extraction residue in the container 1 may be analyzed or further processed. The distilled-off solvent 2 is securely accommodated in the space 13. A new container 1 with a new sample but being empty of solvent can thereupon be connected to the condenser 4, and the solvent in the space 13 may thereafter be released into the container 1 in that the conduit is rotated at its upper end so that it moves upwardly, out of engagement with the bushing 7, that is to say from the lower position in Fig. 2 to the upper position in Fig. 1. The design of the bushing 7 with such a conical inner wall as tapers all the way from the outer side wall of the bushing will give the result that substantially all of the recovered solvent may run into the container 1.

It should be obvious to a person skilled in the art that the illustrated embodiment may be modified such that the bushing 7 could be disposed in the container 1 instead of in the condenser 4, the lower region of the outer wall of the space 13 being then defined by a portion of the lateral defining wall of the container 1.



CLAIMS

1. An apparatus for the recovery of solvent from a solution such as extract, by distillation of the solution and condensation of the solvent vapour by means of a recycle cooler or condenser, and for storing the recovered solvent, c h a r a c t e r i s e d by a
5 conduit (10) for conveyance of the solvent vapour, said conduit (10) being axially movably disposed in and communicating with a space (13) outside the conduit, said space including the condensation surfaces (8) of
10 the condenser (4) and being, at a point downstream of the outlet (12) for the vapour in the space (13), sealable against vapour condensate loss by means of a member (10a) disposed on the outer wall of the conduit (10).
- 15 2. The apparatus according to claim 1, c h a r a c t e r i s e d in that the conduit (10) is defined by a tube which has, a sealed operating end, grippable outside the condenser (4), and orifices (12) in its wall for said communication, said orifices being surrounded
20 by the cooling surfaces (8) of the condenser (4).
3. The apparatus according to claim 1 or 2, c h a r a c t e r i s e d in that a resilient sealing bushing (7) is disposed at said sealing point in the space (13) through which bushing the conduit (10) may
25 be passed for sealing-off the space (13) at said point.
4. The apparatus according to any one of claims 1-3, c h a r a c t e r i s e d in that the condenser (9) is a Liebig, ball or spiral cooler.
5. The apparatus according to any one of claims
30 1-4, c h a r a c t e r i s e d in that the space (13) is defined by the conduit (10) and the cooling surfaces (8) of the condenser.
6. The apparatus according to any one of claims 1-5, c h a r a c t e r i s e d in that the space (13) is

partially defined by a portion of the casing surface of a container (1) for the solution, which container is sealingly but releasably connectible to the condenser (4) and contains said sealing point.

5 7. Use of the apparatus according to any one of claims 1-6 for extraction with hot solvent and/or with solvent vapour and vapour condensate, in particular in the analysis of extracted residue of a sample accommodated in a filter crucible or thimble (5) and located
10 in a container (1) for hot solvent, to which container (1) the apparatus may be connected with vertical orientation.

 8. A method of recovering solvents from a solution, such as extract, by the distillation of the solution
15 and condensation of the solvent vapour, and of storing the recovered solvent, c h a r a c t e r i s e d in that the solvent vapour is led to a condensation space by means of a conduit axially shiftable in the space, said space being closed to the departure of
20 vapour condensate in a direction towards the vapour flow by a movement of the conduit.

FIG.1

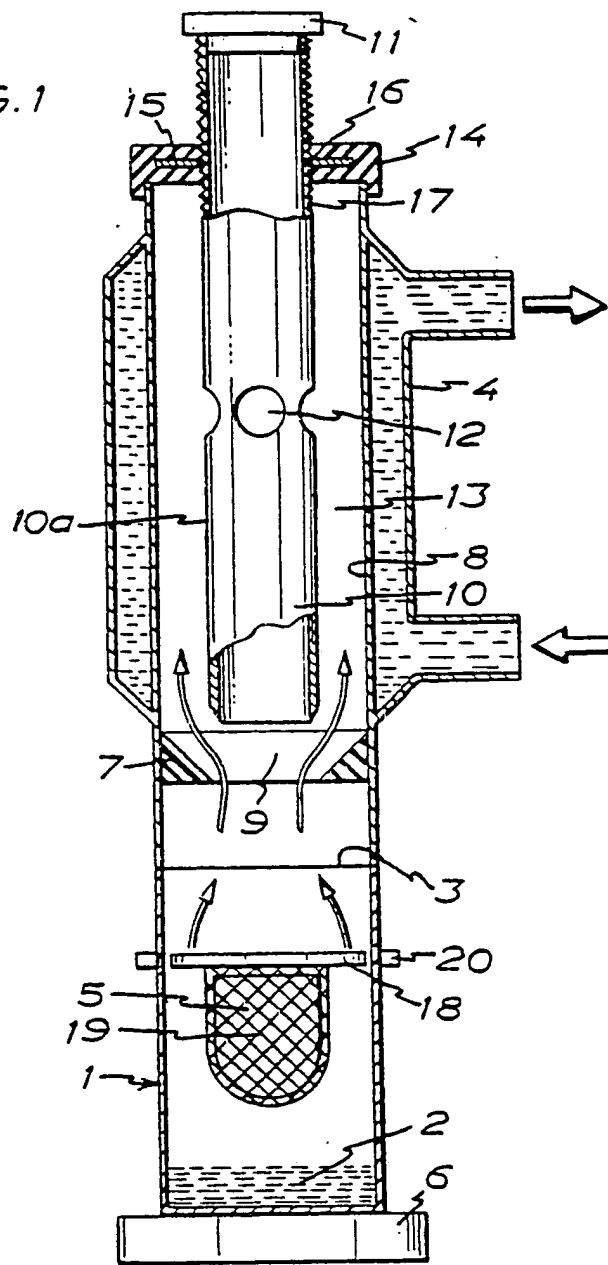
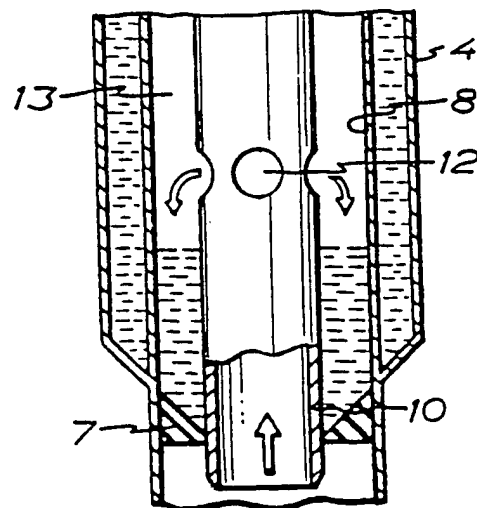
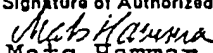


FIG.2



INTERNATIONAL SEARCH REPORT

International Application No PCT/SE79/00054

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ¹		
According to International Patent Classification (IPC) or to both National Classification and IPC B 01 D 5/00, 11/00; B 01 L 11/00		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁴		
Classification System	Classification Symbols	
IPC ²	B 01 D 3/00, 3/40, 5/00, 11/00, 11/02; B 01 J 1/00; B 01 L 11/00; G 01 N 1/28 .../...	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵		
SE; DK, FI, NO classes as above		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴		
Category ⁶	Citation of Document, ¹⁰ with Indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸
A	DE, C, 390 685 published 1924, February 28, Wilhelm Pollmann	
A	DE, C, 507 360 published 1930, September 4, Dr Schalwa Zinzadze	
A	DE, C, 519 371 published 1931, February 5, Arnold Fliedner	
A	DE, C, 858 155 published 1952, December 4, Karl Pietke	
A	GB, C, 899 387 published 1959, August 19, Edmund John Mooney	
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>• Special categories of cited documents: ¹⁵</p> <p>"A" document defining the general state of the art</p> <p>"E" earlier document but published on or after the International filing date</p> <p>"L" document cited for special reason other than those referred to in the other categories</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> </div> <div style="width: 45%;"> <p>"P" document published prior to the International filing date but on or after the priority date claimed</p> <p>"T" later document published on or after the International filing date or priority date and not in conflict with the application, but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search ³		Date of Mailing of this International Search Report ¹
1979-10-31		1979-11-26
International Searching Authority ¹		Signature of Authorized Officer ¹⁹
Swedish Patent Office		 Mats Hammar

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

II

Continuation Fields Searched

US CI

23:230, 272, 272.5, 272.6
 202:168-170, 185, 187, 188, 191, 192
 203:43-46, 98, 99
 422:99, 101, 255, 261, 262, 280-282, 291-293,
 298, 299

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE ¹⁰

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers _____, because they relate to subject matter ¹² not required to be searched by this Authority, namely:
2. ☐ Claim numbers _____, because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out ¹³, specifically:

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING ¹¹

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:
3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

Remark on Protest

- ☐ The additional search fees were accompanied by applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.